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IS 388 (1999): Hydroquinone, photographic grade [CHD 5:
Electroplating Chemicals and Photographic Materials]



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IS 388 : 1999
(Reaffirmed 2006)

भारतीय मानक
हाइड्रोक्विनॉन, फोटोग्राफीय ग्रेड – विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
HYDROQUINONE, PHOTOGRAPHIC GRADE —
SPECIFICATION
(Third Revision)

ICS 37.040.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1999

Price Group 4

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This standard was originally issued in 1952 as a tentative standard and subsequently revised in 1963, incorporating modifications in the requirements for hydroquinone content, melting point and iron content. Changes were also made in the methods of sampling and test. The second revision of this standard was brought out in 1972 modifying the requirements for description and solubility in dilute acetic acid.

In this revision, requirement for heavy metals has been modified along with the test method. The requirement for 'Sulphated ash' has been substituted by 'residue on ignition' and the relevant test method has also been incorporated. An alternate method for hydroquinone determination has also been incorporated.

Composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

4.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained, from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Annex B.

5.2 Criteria for Conformity

A lot shall be declared as conforming if the characteristics tested on the test sample as detailed in **B-4.2** satisfy the requirements given in this specification.

6 QUALITY OF REAGENTS

6.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

ANNEX A

(Clause 3.2)

METHODS OF TEST FOR HYDROQUINONE, PHOTOGRAPHIC GRADE**A-1 DETERMINATION OF HYDROQUINONE**

A-1.0 Two methods have been prescribed namely, Method A and Method B. In case of dispute, Method A shall be the referee method.

A-1.1 Method A**A-1.1.1 Reagents****A-1.1.1.1 Standard ammonium ceric nitrate solution**

Mix about 50 g of ammonium ceric nitrate with 27 ml of concentrated sulphuric acid in a 600-ml beaker, with stirring. Cautiously add water in 100-ml portions, with stirring, allowing 2 to 3 minutes between each addition. Continue adding water until the ceric nitrate is completely dissolved. Dilute to 1 000 ml with water and mix well.

A-1.1.1.2 Arsenic trioxide — Resublimed.**A-1.1.1.3 Sodium hydroxide solution** — 10 percent.**A-1.1.1.4 Dilute sulphuric acid** — 1:5 (v/v) and 0.1 N.**A-1.1.1.5 Osmium tetroxide solution**

Dissolve 0.25 g of osmium tetroxide in 100 ml of approximately 0.1 N sulphuric acid.

CAUTION — Osmium tetroxide is poisonous and contact with it shall be avoided.

A-1.1.1.6 Ferroin indicator solution

Dissolve 1.48 g of *o*-phenanthroline in 100 ml of ferrous sulphate solution (0.025 N).

A-1.1.1.7 Diphenylamine indicator solution

Dissolve 1 g of diphenylamine in 100 ml of concentrated sulphuric acid.

A-1.1.2 Procedure**A-1.1.2.1 Standardization of ammonium ceric nitrate solution**

Weigh accurately about 0.2 g of arsenic trioxide in a watch glass. Transfer the contents of the watch glass to a 250-ml conical flask. Add 15 ml of sodium hydroxide solution and warm gently. When dissolution is complete, cool to room temperature and add 25 ml of dilute sulphuric acid (1:5, v/v). Dilute to 100 ml. Add as a catalyst 0.15 ml of osmium tetroxide solution (*see* Note) and one drop of ferroin indicator solution. Titrate with ammonium ceric nitrate solution until the reddish-orange colour changes to colourless or very pale blue.

$$\text{Normality of ammonium ceric nitrate solution} = \frac{M}{0.04945 V}$$

where

M = mass in g of arsenic trioxide taken, and

V = volume in ml of ammonium ceric nitrate solution used in the titration.

NOTE — Osmium tetroxide solution deteriorates on standing which results in not giving a good end point. In such a case, up to 0.7 ml of the solution may be required.

A-1.1.2.2 Weigh accurately about 0.25 g of the material in a watch glass. Transfer the contents of the watch glass to a 250 ml conical flask containing 100 ml of water and 10 ml of dilute sulphuric acid (0.1 N). Dissolve the material by shaking and add three drops of diphenylamine indicator solution. Titrate with standard ammonium ceric nitrate solution to a red-violet end point.

A-1.1.3 Calculation

$$\text{Hydroquinone, percent by mass} = 5.5 \frac{V N}{M}$$

where

V = volume in ml of standard ammonium ceric nitrate solution used in the titration in **A-1.1.2.2**,

N = normality of standard ammonium ceric nitrate solution as obtained in **A-1.1.2.1**, and

M = mass in g of the material taken for the test in **A-1.1.2.2**.

A-1.2 Method B

A-1.2.1 Reagents

A-1.2.1.1 Concentrated sulphuric acid

A-1.2.1.2 Sulphuric acid — 2 N.

A-1.2.1.3 Dilute ceric sulphate solution

Dissolve 55 g of dry ceric ammonium nitrate in excess (48 ml) of concentrated sulphuric acid in a porcelain dish and evaporate almost to dryness. Dissolve the resulting ceric sulphate in 2 N sulphuric acid. Filter into a 1 000 ml volumetric flask and make up to the mark with 2 N sulphuric acid. Shake well and standardize.

A-1.2.1.4 Ferrous sulphate — A R Grade.

A-1.2.1.5 Ferriin indicator solution (1, 10 phenanthroline-ferrous complex)

Dissolve 0.7 g of ferrous sulphate in 70 ml of water and add 15 g of 1,10 phenanthroline and dilute with water to 100 ml.

A-1.2.1.6 Barium diphenylamine sulphonate

Dissolve 0.3 g of barium diphenylamine sulphonate in 100 ml of water.

A-1.2.2 Procedure

A-1.2.2.1 Ferrous ammonium sulphate solution 0.1 N.

Weigh 40 g of ferrous ammonium sulphate, dissolve in water and add 20 ml of concentrated sulphuric acid. After cooling dilute to 1 000 ml with water.

A-1.2.2.2 Standardization of ferrous ammonium sulphate solution

Pipette out 25 ml of 0.1 N of potassium dichromate solution in a conical flask. Add 25 ml of 5 N sulphuric acid and a few drops of barium diphenylamine sulphonate indicator, and titrate with the ferrous ammonium sulphate solution to a green colour.

A-1.2.2.3 Standardization of ceric sulphate solution

Pipette out exactly 25 ml of 0.1 N ferrous ammonium sulphate in a conical flask. Add 25 ml of 5 N sulphuric acid and a few drops of ferriin indicator. Titrate this solution against ceric sulphate solution to a green end point.

$$\text{Normality of ceric sulphate solution} = \frac{25 \times N}{V}$$

where

N = normality of ferrous ammonium sulphate, and

V = volume in ml of ceric sulphate solution used in the titration.

A-1.2.2.3 Weigh accurately about 0.2 g of the material in a watch glass. Transfer the contents of the watch glass to a 250 ml conical flask and dissolve in 75 ml of water. Add 25 ml of 7 N sulphuric acid and three drops of ferriin indicator. Titrate with 0.1 N ceric sulphate solution to a yellow green end point.

A-1.2.3 Calculation

$$\text{Hydroquinone, percent by mass} = 5.5 \frac{V N}{M}$$

where

V = volume in ml of standard ammonium ceric nitrate solution used in the titration in **A-1.1.2.2**.

N = normality of standard ammonium ceric nitrate solution as obtained in A-1.1.2.1, and

M = mass in g of the material taken for the test in A-1.1.2.2.

A-2 DETERMINATION OF MELTING POINT

A-2.1 Apparatus

A-2.1.1 Glass Heating Vessel — of suitable construction and capacity.

A-2.1.2 Stirrer — capable of rapidly mixing the liquid.

A-2.1.3 Thermometer

The dimensions of the thermometer shall be as follows:

Range	85°C to 215°C
Immersion	100 mm
Stem diameter	5.5 mm to 6.5 mm
Bulb diameter	Not greater than that of stem
Overall length, <i>Max</i>	310 mm
Length of graduated portion, <i>Min</i>	160 mm
Distance of lowest division from bottom of bulb	105 to 115 mm
Accuracy	±0.2°C

A-2.1.4 Capillary Glass Tubes

Made of soft glass, closed at one end, with a wall thickness of 0.10 to 0.15 mm, of sufficient length to allow the open end to be above the surface of the liquid in the heating tube, and of internal diameter 0.8 to 1.2 mm.

A-2.1.4.1 The tubes should preferably be kept sealed at both ends and cut when required.

A-2.2 Procedure

Dry a small quantity of the finely powdered material in a vacuum desiccator. Transfer a portion of the material to a dry capillary glass tube and pack the powder by tapping over hard surface so as to form a tightly packed column, 2 to 4 mm in height. Heat a suitable liquid in the heating vessel and prior to the introduction of the capillary tube, regulate the rate of rise of temperature to 1°C per minute, stirring constantly. When the temperature reaches 160°C, insert the capillary tube, attaching it to the thermometer so that the closed end

is near the middle of the bulb of the thermometer. Adjust the thermometer so that the immersion mark is at the level of the liquid. Note the temperature at which liquefaction of the material occurs, as indicated by the formation of a definite meniscus. Regard this temperature as the melting point of the material.

A-3 DETERMINATION OF RESIDUE ON IGNITION

A-3.1 Apparatus

A-3.1.1 Platinum Crucible

A-3.1.2 Muffle Furnace — capable of maintaining a temperature of 600 ± 50°C.

A-3.2 Procedure

Weigh to the nearest 0.1 g, 5 g of the material into the previously weighed platinum crucible and heat carefully. Finally ignite the residue in the furnace, controlled at 600 ± 50°C, for 4 hours. Cool in a desiccator and weigh the crucible and weigh to the nearest 1 mg. Retain this residue for the heavy metal determination under A-6.

A-3.3 Calculation

$$\text{Residue on ignition, \%} = \frac{M_1 - M_i}{M_2 - M_i} \times 100$$

percent by mass

where

M_1 = mass in g of the crucible,

M_2 = mass in g of the crucible and the material taken for the test, and

M_3 = mass in g of the crucible and residue.

A-4 TEST FOR SULPHATES

A-4.1 Apparatus

A-4.1.1 Nessler Tubes — 50 ml capacity.

A-4.2 Reagents'

A-4.2.1 Dilute Hydrochloric Acid — approximately 1 N.

A-4.2.2 Barium Chloride Solution — 10 percent.

A-4.2.3 Standard Sulphate Solution

Dissolve 1.814 g of potassium sulphate in water and make up the solution to 1 000 ml. Pipette out 10 ml of

this solution into a 100 ml measuring flask and dilute to the mark. One millilitre of the solution contains 0.1 mg of sulphate (as SO_4).

A-4.3 Procedure

Accurately weigh 1.00 g of the material and dissolve in 20 ml of water. Add 1 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Make up the solution to 50 ml. Carry out a control test using 7.5 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume. Compare the turbidity in the two Nessler tubes 15 to 20 minutes after the addition of the barium chloride solution.

A-4.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-5 TEST FOR IRON

A-5.1 Apparatus

A-5.1.1 *Nessler Tubes* — 50 ml capacity.

A-5.1.2 *Concentrated Hydrochloric Acid* — conforming to IS 265.

A-5.1.3 *Ammonium Persulphate*

A-5.1.4 *Butanolic Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake until the solution is clear.

A-5.1.5 *Standard Iron Solution*

Dissolve 7.02 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water. Add a few millilitres of dilute sulphuric acid and dilute to 1 000 ml. When required for use, further dilute 10 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-5.2 Procedure

Weigh accurately 1.00 g of the material into a platinum or silica crucible and ignite at dull-red heat until all the organic matter has been destroyed. Cool the residue and then dissolve in 0.5 ml of concentrated hydrochloric acid. Make up the solution to about 20 ml and add to it 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow the layers to separate. Carry out a control test in another Nessler tube using 2 ml of standard iron solution in place of the material and the

same quantities of other reagents. Compare the colour of the butanol layer in the two tubes.

A-5.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour of the butanol layer in the test with the material is not greater than that produced in the control test.

A-6 TEST FOR HEAVY METALS

A-6.1 Reagents

A-6.1.1 *Hydrochloric Acid* — Relative density approximately 1.18.

A-6.1.2 *Dilute Hydrochloric Acid* — 1 percent.

A-6.1.3 *Dilute Ammonia Solution* — 10 percent.

A-6.1.4 *Heavy Metals Standard Solution*

Dissolve a soluble lead salt in water to give a solution containing 10 mg of lead per 1 000 ml.

A-6.1.5 *Water* — Saturated at room temperature with hydrogen sulphide.

A-6.1.6 *p-Nitrophenol Indicator Aqueous Solution* — 0.25 percent.

A-6.2 Apparatus

A-6.2.1 *Nessler Cylinders* 50 ml capacity.

A-6.3 Procedure

Dissolve the residue obtained from the ignition test under **A-4** in 0.5 ml of dilute hydrochloric acid, warm if necessary, and transfer the solution (with washing) to a beaker using about 40 ml of water. Also take 4 ml of the standard heavy metals solution (**A-6.1.4**) and treat this and the test solution in the following manner. Add 1 drop of the *p*-nitrophenol indicator solution (**A-6.1.6**) and then add drop by drop, the ammonia solution (**A-6.1.3**) until just yellow followed by dilute hydrochloric acid (**A-6.1.2**) drop by drop until colourless again. Add 2.5 ml in excess.

Dilute the test solution to 50 ml with water and transfer a 20 ml aliquot to one of the Nessler cylinders (**A-6.2.1**). Transfer the treated standard heavy metals solution to the other Nessler cylinder and treat both solutions in the following manner. Add 5 ml of the hydrogen sulphide water (**A-6.1.5**), dilute to 50 ml and mix well.

A-6.3.1 The material shall be taken to have passed the test if the colour of the solution in the test with the material is not darker than that of the blank.

ANNEX B
(Clause 5.1)
SAMPLING

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place. Precautions shall be taken to ensure that the material being sampled and the samples are free from adventitious contamination.

B-1.2 The sampling instrument shall be made of glass and the containers for the samples made of brown coloured glass.

B-1.3 The capacity of the containers for the samples shall be such that they are almost completely filled by the sample.

B-1.4 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from ambient temperature and as far as possible, the material shall be protected from light.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In any consignment, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Scale of Sampling

Lot Size	No. of Containers to be Selected
N	n
(1)	(2)
Up to 15	3
16 " 40	4
41 " 65	5
66 " 110	7
111 and above	10

B-2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, random number tables (see IS 4905) shall be used. In its absence, the following procedure of sampling is recommended:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3,..... up to r and so on, where r is the integral part of N/n . Every r th container thus counted shall be withdrawn to give sample for tests.

B-3 PREPARATION OF TEST SAMPLE

B-3.1 From each of the containers selected according to **B-2.3**, a small representative portion of the material not less than 25 g shall be drawn. The portions so drawn from the different containers shall be mixed together, and by the process of conning and quartering an ultimate test sample of about 75 g shall be obtained. This test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.2 All the test samples shall be transferred to separate containers and shall be sealed and labeled with full identification particulars. The referee test sample, bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

B-4 CRITERIA FOR CONFORMITY

B-4.1 Tests for the determination of all the requirements shall be performed on the test sample as obtained in **B-3.1**.

B-4.2 The lot shall be declared as conforming to the requirements of the specification if all the test results as obtained under **B-4.1** satisfy the requirements prescribed.

ANNEX C

(Foreword)

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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